



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/EP95/01892 <b>(22) International Filing Date:</b> 17 May 1995 (17.05.95)  <b>(30) Priority Data:</b> 9410092.2                      19 May 1994 (19.05.94)                      GB  <b>(71) Applicant (for all designated States except US):</b> KELCO INTERNATIONAL LIMITED [GB/GB]; Waterfield, Tadworth, Surrey KT20 5HQ (GB).  <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> FITZPATRICK, John [GB/GB]; 31 Treelands, North Holmwood, Surrey RH5 4TE (GB). MELLOR, Adrian [GB/GB]; 11 Camelot Close, Southwater, West Sussex RH13 7XP (GB).  <b>(74) Agents:</b> BOSCH, Henry et al.; Monsanto Services International S.A./N.V., Patent Dept., Avenue de Tervuren 270-272 P.O. Box 21, B-1150 Brussels (BE).		<b>(81) Designated States:</b> JP, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> EMULSION COMPRISING A GELLED AQUEOUS OUTER PHASE, A NON-AQUEOUS INTERMEDIATE PHASE AND AN AQUEOUS INNER PHASE  <b>(57) Abstract</b> <p>The present invention relates to an emulsion comprising an aqueous gelled outer phase, a non-aqueous intermediate phase and an aqueous inner phase containing an active ingredient incompatible with the gelled outer phase, wherein the inner phase containing the active ingredient is encapsulated by the intermediate phase. Said emulsion finds suitable application in an antiperspirant, as a sustained release drug or personal hygiene products.</p>		

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**EMULSION COMPRISING A GELLED AQUEOUS OUTER PHASE, A NON-AQUEOUS INTERMEDIATE PHASE AND AN AQUEOUS INNER PHASE**

The present invention is concerned with an emulsion, a method of preparing the same and its use, and in particular an emulsion comprising a gelled outer phase having an  
5 active ingredient dispersed therein.

Encapsulation of active ingredients in gels, such as gellan gum, is well known. For example, JP 62125850 discloses encapsulation of ingredients, such as food, oils, medicines and the like, within beads of gellan  
10 gum. In an example, a salad oil emulsion was added as 0.5ml size drops to a 1% gellan gum solution. The resulting beads had a 0.35mm thick skin and contained 0.3ml of oil in each sphere.

US4563366 discloses a gelled food product which  
15 comprises a matrix containing at least one dispersed food ingredient which comprises vegetable, fruit, meat, fish, sugar, and/or milk.

GB2219803 discloses a gelling composition which comprises a blend of gellan, kappa-carrageenan and mannan.  
20 The gelling composition is useful as a gelling matrix in food products such as pet foods and the like.

JP 63267361 discloses a gel which can contain fragrances, microbicides, insecticides, and the like, in addition to a gelation agent selected from gellan gum, and  
25 its combination with carrageenan, gelatin, agar, locust bean gum, xanthan gum, carboxymethyl cellulose and the like.

Incorporation of certain active ingredients in gel matrices comprising anionic hydrocolloids, such as gellan gum, has however proved to be problematic due to the  
30 incompatibility of the active ingredients with the gels causing degradation or precipitation of the latter.

This incompatibility can be seen, for example, when aluminium chlorohydrate is blended with gellan gum in an

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attempt to prepare an antiperspirant, whereby undesirable precipitation of the gellan gum occurs. Similarly, the incompatibility of other cationic ingredients, such as cationic drugs (verapamil hydrochloride, chloropheniramine  
5 maleate and the like) and cationic surfactants (such as benzalkonium chlorides and the like) with the above-mentioned anionic gel matrices has proven to be problematic.

The present invention alleviates the above problem, wherein active ingredients incompatible with a gel matrix  
10 can be incorporated therein without adversely affecting the properties of the gel.

According to the present invention there is provided an emulsion comprising an aqueous gelled outer phase, a non-aqueous intermediate phase and an aqueous inner phase  
15 containing an active ingredient incompatible with the gelled outer phase, wherein the inner phase containing the active ingredient is encapsulated by the intermediate phase.

An emulsion according to the present invention therefore alleviates the problem described above, in that the  
20 intermediate phase separates the aqueous gelled outer phase from the active ingredients present in the aqueous inner phase.

Aptly the gelled outer phase comprises one or more gelled anionic hydrocolloids. A frequently employed  
25 hydrocolloid in the present invention is gellan gum, other suitable hydrocolloids being alginates, pectins, carrageenans, agar, locust bean gum and the like.

Gellan gum refers to the extracellular polysaccharide obtained by the aerobic fermentation of the microorganism,  
30 Pseudomonas elodea, in a suitable nutrient medium. Various forms of gellan gum are known e.g., native, deacetylated, deacetylated clarified, partially deacetylated, and partially deacetylated clarified.

It is preferred that the gellan gum employed in the  
35 present gel is a "low acetyl" gellan gum. As used herein,

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the term "low acetyl" denotes a level of acylation of the gellan gum of 0.3 to 0% by weight.

Various alginates useful in this invention are described in detail by I.W. Cottrell and P. Kovacs in  
5 "Alginates," as Chapter 2 of Davidson, ed., Handbook of Water-Soluble Gums and Resins (1980).

Alginates include "bioalgin" and "algal" alginate.

Biolalgin is microbially produced polysaccharides produced by both Pseudomonas and Azotobacter Strains as  
10 described, for example, in Jarman et al., United States patent 4,235,966. These alginates are polysaccharides consisting of a partially acetylated variable block copolymer of D-mannuronic and L-guluronic acid residues. Jarman et al. state that the polysaccharide produced is  
15 similar to that produced from seaweed except that the molecule is partially acetylated.

The term "algal" alginate refers to naturally derived alginic acid and salts thereof. Naturally derived alginic acid, derived primarily from kelp, is a commercially  
20 available product, e.g., KELACID™ (Kelco Div., formerly Merck & Co., Inc., now acquired by Monsanto Company). The salts include appropriate metal salts, e.g. alkali metal, alkaline earth metal, ammonium salts, and organic derivatives, e.g. alkylene glycol, propylene glycol and the  
25 like. The preferred salts are sodium, potassium, magnesium, ammonium and propylene glycol algal alginate. Most preferred herein are naturally derived algal sodium alginates, such as those sold commercially under the trademarks KELTEX, KELGIN and KELTONE™ by Kelco Division formerly Merck & Co., Inc.,  
30 now Monsanto Company.

Pectins are plant cell wall polysaccharides comprising branched molecules that contain many negatively charged galacturonic acid residues. In view of their negative charge pectins are highly hydrated and readily bind to cations so

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as to be suitable for forming the gelled outer phase of the emulsion of the present invention.

Locust bean gum is an extract of the locust bean or carob, Ceratonia siliqua. It is commercially available and is often used as a stabilizer in foods such as ice cream, sausages, and cheese.

It is preferred that the anionic hydrocolloid is present in the gelled outer phase in an amount of 0.1 to 5% by weight, based on the weight of the outer phase, for example 0.25 to 2.5% by weight. In the case where an abradable gelled outer phase is required, for example in the case where the emulsion is for use as an antiperspirant, the hydrocolloid is typically present in an amount of 0.75 to 1.25% by weight, based on the weight of the outer phase. Alternatively for applications such as drug release systems, wherein the gelled phase is required to be erodible when in contact with body fluids, the anionic hydrocolloid is typically present in an amount of 0.5 to 0.6% by weight of the outer gelled phase.

Optionally the outer phase may, in some applications of the emulsion according to the present invention, such as for antiperspirants, body lotions and the like, further contain a fragrance which may typically be present in an amount of 0.75 to 1.25% by weight, based on the weight of the outer phase. The outer phase may contain colourant if desired.

The gelled outer phase may also optionally contain a preservative, a preferred preservative being n-propyl p-hydroxybenzoate. The preservative is suitably employed in a minor amount, such as not greater than about 0.2% by weight of the gelled outer phase.

Optionally the gelled outer phase may further contain a biocide, typically present in an amount of 0.05 to 2.5% by weight based on the weight of the outer phase.

Suitably one or more surfactants are also included in the outer phase, examples of suitable surfactants comprising

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diethanolamide cetyl phosphate, polyoxyethylene castor oil, polyoxyethylene hydrogenated castor oil, polyoxyethylene triglycerides, polyoxyethylene lanolin, polyoxyethylene laurates, polyoxyethylene stearates and the like. Generally  
5 the surfactant is present in the outer phase at a level of 1 to 3% by weight, based on the weight of the outer phase, more preferably the surfactant is present at a level of 1.5 to 2.5% by weight, based on the weight of the outer phase.

The anionic hydrocolloid may typically be gelled by a  
10 suitable cation such as calcium, magnesium or the like.

In the case of such multivalent gelling cations, these cations are suitably provided by salts such as calcium chloride, magnesium chloride, calcium sulphate, magnesium sulphate and the like. The gelling multivalent cations are  
15 generally present in the outer phase of the emulsion at a level not greater than 0.05% by weight, based on the weight of the outer phase.

In the case where the anionic hydrocolloid comprises gellan gum, it is preferred that monovalent cations such as  
20 sodium, potassium and the like are employed to gel the gellan gum, potassium being particularly preferred. Aptly the monovalent cations are provided by a suitable salt such as sodium chloride, potassium chloride, trisodium citrate, tripotassium citrate and the like; in the case of potassium a  
25 preferred salt is tripotassium citrate.

Advantageously the gelling monovalent cations are present in the outer phase at a level not greater than 0.5% by weight, based on the weight of the outer phase.

In the case where monovalent gelling cations are  
30 employed it is preferred that the outer phase is essentially free of multivalent ions, such as calcium, magnesium or the like. The skilled worker will appreciate that it is most unusual to avoid gelling quantities of multivalent ions in this way since it is normal practice  
35 in the art to use multivalent ions to increase gel

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strength.

Desirably the non-aqueous intermediate phase comprises an oil phase which can comprise volatile silicone oils, petroleum, paraffin, or vegetable oil such as olive oil, arachis oil, castor oil, cottonseed or rapeseed oil or the like. In this way, there is provided by the present invention a triple phase emulsion of a water in oil emulsion dispersed in a water phase. A favoured oil phase in the present invention comprises a silicone oil, preferably a volatile silicone oil, although it is of course appreciated that the other above-mentioned oils can similarly be employed in the intermediate phase.

As hereinbefore described, an aim of the present invention is to encapsulate within a non-aqueous phase, an inner aqueous phase containing active ingredients incompatible with the gelled outer phase.

Aptly the active ingredients comprise cationic materials which are incompatible with anionic hydrocolloid gels in that the former would effect degradation or precipitation of the latter.

Examples of such cationic materials include polyvalent metal ions, typically present as salts such as aluminium and/or zirconium salts, typically aluminium chlorohydrate, aluminium-zirconium chlorohydrate and the like, cationic drugs such as verapamil hydrochloride, chloropheniramine maleate and the like, and cationic surfactants such as benzalkonium chlorides, cetyl trimethyl ammonium chloride, lauryl dimethyl ammonium chloride and the like.

It can be appreciated from the above range of active cationic ingredients that an emulsion according to the present invention has several applications.

In a first embodiment wherein the active cationic ingredient includes aluminium ions, the emulsion according to the present invention has applications as an antiperspirant; such antiperspirants containing aluminium



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ions were not previously prepared because of the incompatibility of aluminium containing materials with gels such as the anionic hydrocolloids suitable for use in the present invention.

5 In a second embodiment of the present invention wherein cationic drugs are present in the aqueous inner phase encapsulated by the intermediate oil phase, the emulsion of the present invention is useful as a sustained release drug system wherein the outer gel phase is slowly erodible when in  
10 contact with bodily fluids so as to release the encapsulated drugs.

In a further embodiment of the invention wherein the active ingredients comprise cationic surfactants, personal hygiene products, such as body lotions, moisturisers,  
15 creams and the like, can be provided by the present invention. The provision of such personal hygiene products employing an emulsion according to the present invention is desirable in that a combination of the well known moisturising properties of water in oil emulsions  
20 and the desirable skin feel properties of oil in water emulsions is achieved.

Aptly the inner phase comprises an aqueous solution of the active ingredient, wherein the solvent typically comprises water. The active ingredient is desirably  
25 present up to its limit of solubility, and in the case where aluminium chlorohydrate (which is a favoured ingredient) is employed as the active ingredient the former can be included in an amount of up to 80% by weight, based on the weight of the inner phase. It can be  
30 appreciated that the inclusion of the active ingredient up to its limit of solubility in the solvent of the aqueous phase is beneficial in allowing quantities of the active ingredient, which would otherwise be incompatible with the gelled outer phase, to be incorporated in the emulsion of  
35 the present invention.

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Preferably a primary emulsion is initially formed wherein the aqueous solution of the active ingredient is dispersed within the non-aqueous phase. Desirably 45 to 85% by weight of the aqueous solution is dispersed in 15 to 55%  
5 by weight of the non-aqueous phase. Suitable 55 to 85% by weight of the primary emulsion is subsequently dispersed within a percentage balance by weight of an aqueous dispersion of a gellable material, such as gellan gum or the like.

10 A hydrophobic surfactant is generally included in the non-aqueous phase. Examples of suitable surfactants include cetyl dimethicone copolyol, polysorbate 60, sorbitan monolaurate, sorbitan monostearate, sorbitan mono-oleate, sorbitan monopalmitate, sorbitan trioleate, polyethylene  
15 glycol-6-sorbitan beeswax, polyethylene glycol-20-sorbitan beeswax, ceteth-20-stearate, ceteth-2-stearate, ceteth-20-oleate, aluminium magnesium hydroxide stearate and the like. Typically the surfactant is present at a level of 0.5 to 2.5% by weight, based on the weight of the non-aqueous  
20 phase. In the case where polysorbate 60 is employed, this surfactant is generally included at a level of 1.5 to 2.5% by weight, whereas cetyl dimethicone copolyol is typically employed at a level of 0.5 to 1.5% by weight.

A particularly preferred emulsion according to the  
25 present invention comprises an outer gelled phase comprising gellan gum, typically gelled by monovalent ions substantially as hereinbefore described, an intermediate phase comprising silicone oil and an aqueous inner phase containing an active cationic ingredient  
30 particularly aluminium chlorohydrate or aluminium-zirconium chlorohydrate. This particularly preferred emulsion is suitable for use as antiperspirant and there is further provided by the present invention an anti-perspirant which comprises an emulsion comprising an outer  
35 gelled phase comprising gellan gum, an intermediate phase

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comprising silicone oil and an aqueous inner phase containing an active cationic ingredient selected from the group consisting of aluminium chlorohydrate and aluminium-zirconium chlorohydrate, wherein the inner phase containing the active  
5 cationic ingredient is encapsulated by the intermediate phase.

An anti-perspirant according to the present invention is generally a "stick" type anti-perspirant, whereby the emulsion is substantially solid. A "stick" type anti-  
10 perspirant as described herein typically comprises a substantially solid body of an emulsion according to the present invention configured to be received within a container, whereby the body is movable relative to the container between advanced and retracted positions.

There is still further provided by the present invention use of an anionic hydrocolloid substantially as hereinbefore described to provide a gelled outer phase of an emulsion, wherein the emulsion comprises a gelled outer phase comprising the anionic hydrocolloid, an intermediate non-  
20 aqueous phase and an aqueous inner phase containing an active ingredient incompatible with the gelled outer phase, whereby the inner phase containing the active ingredient is encapsulated by the intermediate phase.

There is further provided by the present invention a  
25 method of preparing an emulsion substantially as hereinbefore described, which method comprises dispersing, in a non-aqueous phase, an aqueous phase containing an active ingredient, so as to produce a primary two phase emulsion, mixing the primary emulsion with an aqueous dispersion of a  
30 gellable material and effecting gelation thereof.

Aptly the gellable material comprises one or more anionic hydrocolloids substantially as hereinbefore described, wherein the employ of gellan gum is particularly preferred. Similarly the non-aqueous phase and the active  
35 ingredient are substantially as hereinbefore described,

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wherein preferably the non-aqueous phase comprises a volatile silicone oil and the active ingredient comprises a cationic material incompatible with the anionic hydrocolloid.

Desirably a hydrophobic surfactant, such as cetyl dimethicone copolyol as described above, is intimately mixed with the non-aqueous phase prior to dispersal of the first mentioned aqueous phase therein. The employ of such a surfactant is beneficial in achieving stabilisation of the primary two phase emulsion.

Advantageously the non-aqueous phase is initially subjected to relatively low energy and shear agitation, typically employing a stirrer, such as a paddle stirrer or the like, stirring at a speed in the range of 350 to 450 rpm (preferably 390 to 410 rpm), during addition of the aqueous phase thereto.

Subsequently agitation of relatively high energy and shear is employed, typically stirring at a speed in the range of 1400 to 1600 rpm, preferably 1480 to 1520 rpm, whereby a stable primary emulsion is formed.

Typically the primary emulsion is heated to a temperature in the range of 50 to 60°C prior to mixing with an aqueous dispersion of a gellable material.

Suitably the method involves dispersing a gellable material, typically the anionic hydrocolloid as hereinbefore described, in an aqueous carrier such as water, prior to mixing with the primary emulsion. The resulting aqueous dispersion is generally heated to a temperature in the range of 80 to 90°C to effect hydration followed by addition of DEA cetyl phosphate or other similar surfactant as hereinbefore described. Aptly the dispersion is cooled to a temperature in the range of 50 to 60°C prior to mixing with the primary emulsion. Optionally the mixture achieved on mixing the primary emulsion with the aqueous dispersion may be subjected to further heating to a temperature in the range of 65 to 70°C.

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Gelation is desirably achieved by addition of gelling cations, typically monovalent ions such as potassium or sodium in the case where gellan gum is employed as the anionic hydrocolloid. Aptly the mixture is allowed to cool  
 5 and set to form a gel. According to a preferred aspect of the invention tripotassium citrate is employed.

The weight percentages of the aqueous and nonaqueous phases, gelling cations and the like is substantially as hereinbefore described. Similarly the method may optionally  
 10 further include blending ingredients, such as a fragrance, preservative or the like, as hereinbefore described, with the aqueous dispersion of the gellable material.

The present invention will now be illustrated by the following examples, which are for illustrative purposes only.

#### 15 Example 1

##### Part A

	% w/w
Polysorbate 60	2.0
Aluminium-zirconium chlorohydrate	49.0
20 (50% aqueous solution)	
Silicone oil	49.0

##### Part B

Gellan gum	0.6
Biocide (triclosan)	2.0
25 Deionised water	95.4
Polyethoxylated hydrogenated castor oil (60 ethylene oxide groups)	2.0

Part A was prepared by initially blending the  
 30 polysorbate 60 with the silicone oil. The aluminiumzirconium chlorohydrate was subsequently slowly added (by burette) whilst stirring at 400rpm ( $\pm$  5rpm) using a paddle stirrer,

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followed by stirring at 1500rpm for 3-4 minutes to form a primary water in oil emulsion. The resulting primary emulsion was heated to 55°C prior to intimately mixing with Part B.

5 Part B was prepared by dispersing the gellan gum in water, heating to 85°C to effect hydration of the gellan gum, followed by addition of the polyethoxylated hydrogenated castor oil and biocide thereto and cooling to 55°C for mixing with the primary emulsion of Part A.

10 50 parts by weight of the primary emulsion of Part A were mixed with 50 parts by weight of Part B whilst stirring at 800rpm, followed by heating to 65-70°C. 0.5 parts by weight of  $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$  (0.1M) were added to gel the gellan gum, whereby gelation occurred on cooling.

15 Example 2

Part A

	% w/w
Cetyl dimethicone copolyol	1.0
Silicone oil	20.0
20 Aluminium chlorohydrate	79.0
(50% aqueous solution)	

Part B

Gellan gum	1.0
DEA cetyl phosphate	2.0
25 Biocide (triclosan)	0.6
Deionised water	96.4

Part A was prepared by dissolving the cetyl dimethicone copolyol in the silicone oil. The aluminium chlorohydrate was subsequently slowly added (by burette) whilst stirring at

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400rpm ( $\pm$  5rpm) using a paddle stirrer, followed by stirring at 1500rpm for 3-4 minutes to form the primary water in oil emulsion. The resulting primary emulsion was heated to 55°C prior to intimately mixing with Part B.

5        Part B was prepared by dispersing the gellan gum in water, heating to 85°C to effect hydration of the gellan gum, followed by addition of the DEA cetyl phosphate and the biocide thereto and cooling to 55°C for mixing with the primary emulsion of part A.

10       67 parts by weight of the primary emulsion of Part A were added to 33 parts by weight of Part B, whilst stirring at 800rpm, followed by heating to 65-70°C. Tripotassium citrate was added to gel the gellan gum, whereby gelation occurred on cooling.

Claims

1. An emulsion comprising an aqueous gelled outer phase, a non-aqueous intermediate phase and an aqueous inner phase containing an active ingredient incompatible with the gelled outer phase, wherein the inner phase containing the active ingredient is encapsulated by the intermediate phase.

2. An emulsion according to claim 1, wherein the gelled outer phase comprises one or more gelled anionic hydrocolloids selected from the group consisting of gellan gum, alginates, pectins, carrageenans, agar, and locust bean gum.

3. An emulsion according to claim 2, wherein the anionic hydrocolloid is present in the gelled outer phase in an amount of 0.1 to 5% by weight, based on the weight of the outer phase.

4. An emulsion according to claim 2 or 3, wherein the anionic hydrocolloid comprises gellan gum.

5. An emulsion according to claim 4, wherein the gellan gum is gelled by monovalent cations.

6. An emulsion according to any of claims 1 to 5, wherein the non-aqueous intermediate phase comprises an oil phase comprising silicone oil.

7. An emulsion according to any of claims 2 to 6, wherein the active ingredient comprises a cationic ingredient incompatible with the anionic hydrocolloid of the outer phase.



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8. An antiperspirant comprising an emulsion comprising a gelled outer phase comprising gellan gum, an intermediate phase comprising silicone oil and an aqueous inner phase containing an active cationic ingredient selected  
5 from the group consisting of aluminium chlorohydrate and aluminium -zirconium chlorohydrate, wherein the inner phase containing the active cationic ingredient is encapsulated by the intermediate phase.

9. A sustained release drug system comprising an  
10 emulsion according to any of claims 1-7, comprising a cationic drug in an aqueous inner phase encapsulated by an intermediate oil phase, dispersed in an outer gel phase which is erodible when in contact with body fluids.

10. Use of an emulsion according to any of claims  
15 1-7 as personal hygiene product, wherein the active ingredients comprise cationic surfactants, personal hygiene products, such as body lotions, moisturisers, or creams.

11. A method of preparing an emulsion according to any of claims 1 to 7, which method comprises dispersing, in a  
20 non-aqueous phase, an aqueous phase containing an active ingredient, so as to produce a primary emulsion, mixing the primary emulsion with an aqueous dispersion of a gellable material and effecting gelation thereof.

12. A method according to claim 11, wherein 45 to  
25 85% by weight of an aqueous solution of the active ingredient is dispersed in 15 to 55% by weight of the non-aqueous phase to form the primary emulsion, and 55 to 85% by weight of the primary emulsion is subsequently dispersed within a percentage balance by weight of the aqueous  
30 dispersion of the gellable material.

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 95/01892

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 A61K9/10 A61K9/107 A61K9/113 A61K7/00 A61K7/32  
 A61K7/38 A61K7/48 A61K9/06

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 345 075 (UNILEVER PLC) 6 December 1989 see abstract; claims 1-9 ---	1-12
A	EP,A,0 507 693 (L'OREAL) 7 October 1992 see abstract; claims 1-22 ---	1-12
A	EP,A,0 278 103 (AMERICAN CYANAMID COMPANY) 17 August 1988 see claims 1-9 ---	1-12
A	US,A,4 900 542 (UMBERTO V. PARROTTA ET AL.) 13 February 1990 see the whole document ---	1-12
A	EP,A,0 279 328 (FIRMENICH SA) 24 August 1988 see abstract; claims 1-6 ---	1-12
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Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

## \* Special categories of cited documents :

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# INTERNATIONAL SEARCH REPORT

International Application No

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

International Application No

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		AU-B- 597462	31-05-90
		AU-B- 1017088	14-07-88
		CA-A- 1318587	01-06-93
		DE-A- 3782420	03-12-92
		ES-T- 2052542	16-07-94
		JP-A- 63258425	25-10-88
US-A-4900542	13-02-90	ZA-A- 8800152	28-06-88
		US-A- 4673570	16-06-87
EP-A-279328	24-08-88	CH-A- 675966	30-11-90
		AU-B- 609356	26-04-91
		AU-B- 1196788	25-08-88
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